

THE CLAIMS

What is claimed is:

1. A process for reducing the concentration of water and optionally at least one impurity, from a cyclosiloxane precursor, wherein said process is selected from the group consisting of:

(1) contacting the cyclosiloxane precursor with an adsorbent bed material, so as to remove therefrom at least a portion of the water, and optionally at least one other impurity, to produce a cyclosiloxane precursor having a reduced level of water and optionally impurity; and removing the purified cyclosiloxane precursor from the adsorbent bed material; and

(2) distilling a starting mixture comprising at least water and at least one $[\text{SiO}]_n$ cyclosiloxane CVD precursor, in the presence of an azeotropic component, so as to form an azeotropic mixture with the water contained in said starting mixture; in order to produce (A) a distillate fraction comprising water and the azeotropic component and (B) a balance fraction comprising a purified cyclosiloxane precursor, whereby said balance fraction (B) is substantially reduced in water relative to said starting mixture; and

(3) a combination of 1 and 2.

2. The process according to claim 1, wherein said impurity is selected from the group consisting of acidic and basic impurities.

3. The process according to claim 1, wherein said impurity is acidic.

4. The process according to claim 1, wherein said impurity is basic.

5. The process according to claim 1, wherein said cyclosiloxane precursor comprises the formula $[\text{R R}' \text{ Si-O}]_n$, wherein each of R and R' is same or different and independently selected from the group consisting of hydrogen, hydroxyl, $\text{C}_1\text{-C}_8$ alkyl, $\text{C}_1\text{-C}_8$ alkoxy, $\text{C}_1\text{-C}_8$ alkene, $\text{C}_1\text{-C}_8$ alkyne, and $\text{C}_1\text{-C}_8$ carboxyl; and n is from 2 to 8.

6. The process according to claim 1, wherein the cyclosiloxane precursor is selected from the group consisting of polyhedral oligomeric silsesquioxanes (POSS), octamethylcyclotetrasiloxane (OMCTS), hexamethylcyclotetrasiloxane (HMCTS), tetramethylcyclotetrasiloxane (TMCTS), and mixtures thereof.
7. The process according to claim 1, wherein the cyclosiloxane precursor is 1,3,5,7-tetramethylcyclotetrasiloxane.
8. The process according to claim 2, wherein said process comprises contacting the cyclosiloxane precursor with an adsorbent bed material, so as to remove therefrom at least a portion of the water, and optionally at least one other impurity, to produce a cyclosiloxane precursor having a reduced level of water and optionally at least one other impurity; and removing the purified cyclosiloxane precursor from the adsorbent bed material.
9. The process according to claim 1, wherein said adsorbent bed material is selected from the group consisting of: silica gel, molecular sieves, aluminum oxide, and carbon.
10. The process according to claim 1, wherein said drying agent is selected from the group consisting of: calcium oxide, calcium chloride, sodium sulfate, magnesium perchlorate, phosphorus pentoxide, silicide, metals, and metal hydrides, such as calcium hydride.
11. The process according to claim 1, wherein the drying agent is calcium oxide.
12. The process according to claim 1, wherein the drying agent is calcium hydride.
13. The process according to claim 1, wherein the adsorbent further comprises a second adsorbent or a drying agent.
14. The process according to claim 1, wherein the drying agent further comprises a second drying agent or an adsorbent.
15. The process according to claim 1, wherein said purified cyclosiloxane precursor is removed from said adsorbent and/or drying agent by distillation.

16. The process according to claim 1, wherein said purified cyclosiloxane precursor is removed from said adsorbent and/or drying agent by decantation.
17. The process according to claim 1, wherein said purified cyclosiloxane precursor is removed from said adsorbent and/or drying agent by pump.
18. The process according to claim 2, wherein the level of at least one impurity in the purified cyclosiloxane precursor is reduced to a level of less than $< 0.001\%$.
19. The process according to claim 2, wherein the level of at least one impurity in the purified cyclosiloxane precursor is reduced to a level of less than $< 0.00001\%$.
20. The process according to claim 2, wherein the level of water in the purified cyclosiloxane precursor is reduced to a level of less than $< 0.001\%$.
21. The process according to claim 2, wherein the level water in the purified cyclosiloxane precursor is reduced to a level of less than $< 0.00001\%$.
22. The process according to claim 1, wherein the starting mixture comprising at least water and at least one $[\text{SiO}]_n$ cyclosiloxane CVD precursor, is distilled in the presence of an azeotropic component, so as to form an azeotropic mixture with the water contained in said starting mixture; in order to produce (A) a distillate fraction comprising water and the azeotropic component and (B) a balance fraction comprising a purified cyclosiloxane precursor, whereby said balance fraction (B) is substantially reduced in water relative to said starting mixture.
23. The process according to claim 1, wherein the azeotropic component is selected from the group consisting of: benzene, methanol, ethanol, 2-propanol, butyl acetate, phenol, chloroform, methyl acrylate, isopropyl formate, isopropyl acetate, cyclohexane, and n-heptane.
24. The process according to claim 1, wherein the azeotropic component is benzene.
25. The process according to claim 4, wherein the azeotropic component is benzene.

26. The process according to claim 1 wherein the distillate fraction comprising water and the azeotropic component, and the cyclosiloxane precursor distill at temperatures that are at least 10°C apart.

27. The process according to claim 1 wherein the distillate fraction comprising water and the azeotropic component, and the cyclosiloxane precursor distill at temperatures that are at least 30°C apart

28. The process according to claim 1 wherein the distillate fraction comprising water and the azeotropic component, and the cyclosiloxane precursor distill at temperatures that are at least 50°C apart

29. The process according to claim 1 comprising

(1) contacting the cyclosiloxane precursor with an adsorbent bed material, so as to remove therefrom at least a portion of the at least one impurity, to produce a cyclosiloxane precursor having a reduced level of impurity;

(2) distilling the cyclosiloxane precursor having a reduced level of acidic impurity in the presence of at least one azeotropic component so as to form an azeotropic mixture with the water contained in said starting mixture; in order to produce (A) a distillate fraction comprising water and the azeotropic component and (B) a balance fraction comprising cyclosiloxane, whereby said balance fraction (B) is substantially reduced in water relative to the starting mixture; and

(3) distilling said balance fraction (B) to isolate cyclosiloxane precursor having reduced levels of at least one impurity and water.

30. A CVD method of depositing a low dielectric constant thin film on a substrate from a $[\text{SiO}]_n$ cyclosiloxane precursor that has been purified by a method selected from the group consisting of:

(1) contacting the cyclosiloxane precursor comprising water and optionally at least one impurity with an adsorbent bed material, so as to remove therefrom at least a portion of the water and optionally at least a portion of the impurity, to produce a purified cyclosiloxane

precursor having a reduced level of water and optionally at least one impurity; and removing the purified cyclosiloxane precursor from the adsorbent bed; and

(2) distilling a starting mixture comprising at least water and at least one $[\text{SiO}]_n$ cyclosiloxane CVD precursor, in the presence of an azeotropic component, so as to form an azeotropic mixture with the water contained in said starting mixture; in order to produce (A) a distillate fraction comprising water and the azeotropic component and (B) a balance fraction comprising purified cyclosiloxane precursor, whereby said balance fraction (B) is substantially reduced in water relative to the starting mixture; and

(3) a combination of 1 and 2.

31. The method according to claim 30, said CVD method comprises the steps of:

placing the substrate in a chemical vapor deposition apparatus;

introducing at least one vaporized organosilicon precursor comprising at least one purified cyclosiloxane precursor into the apparatus;

transporting the purified cyclosiloxane precursor vapor into a chemical vapor deposition zone containing a substrate, optionally using a carrier gas to effect such transport;

contacting the purified cyclosiloxane precursor vapor with the substrate under chemical vapor deposition conditions to deposit a thin film comprising an organosilicon composition; and

annealing the organosilicon thin film to produce a porous, SiCOH, low dielectric constant thin film.

32. The method according to claim 30, wherein said CVD method is plasma enhanced.

33. The method according to claim 31, further comprising a co-reactant.

34. The method according to claim 33, wherein the co-reactant is a gas.

35. The method according to claim 33, wherein the co-reactant is a liquid.

36. The method according to claim 33, wherein the co-reactant is an organic species.
37. The method according to claim 33, wherein the co-reactant is selected from the group consisting of: CO₂, ethylene, acetylene, N₂O, O₂, H₂ and mixtures thereof.
38. The method according to claim 33, wherein said co-reactant is selected from the group consisting of, C₁-C₈ alkyl, C₁-C₈ alkoxy, C₁-C₈ alkene, C₁-C₈ alkyne, C₁-C₈ aryl, C₅-C₁₂ aryl and C₁-C₈ carboxyl.
39. The method according to claim 31 further comprising a co-reactant selected from the group consisting of: organosilicon precursors, C₁-C₈ alkyl, C₁-C₈ alkoxy, C₁-C₈ alkene, C₁-C₈ alkyne, C₁-C₈ aryl, C₅-C₁₂ aryl and C₁-C₈ carboxyl, CO₂, ethylene, acetylene, N₂O, O₂, H₂ and mixtures thereof.
40. The method according to claim 31, wherein the inert carrier gas is selected from the group consisting of: argon, helium, and CO₂.
41. The method according to claim 32, wherein the plasma is generated from single or mixed frequency RF power.
42. The method according to claim 32, wherein, PECVD process is tuned with single frequency or dual frequency operating simultaneously to yield the SiCOH thin film.
43. The method according to claim 31, wherein the SiCOH thin film is post annealed in a furnace, at a temperature in the range of from about 100°C to about 400°C.
44. The method according to claim 31, wherein the SiCOH thin film is annealed at a gradually increasing temperature.
45. 1,3,5,7-tetramethylcyclotetrasiloxane purified by a process selected from the group consisting of:

- (1) contacting the cyclosiloxane precursor with an adsorbent bed material, so as to remove therefrom at least a portion of the water, and optionally at least one impurity, selected from the group consisting of acidic and basic impurities, to produce a cyclosiloxane precursor

having a reduced level of water and optionally at least one impurity; and removing the purified cyclosiloxane precursor from the adsorbent bed material; and

(2) distilling a starting mixture comprising at least water and at least one $[\text{SiO}]_n$ cyclosiloxane CVD precursor, in the presence of an azeotropic component, so as to form an azeotropic mixture with the water contained in said starting mixture; in order to produce (A) a distillate fraction comprising water and the azeotropic component and (B) a balance fraction comprising a purified cyclosiloxane precursor, whereby said balance fraction (B) is substantially reduced in water relative to said starting mixture; and

(3) a combination of 1 and 2.

46. A SiCOH low dielectric constant thin film deposited from 1,3,5,7-tetramethylcyclotetrasiloxane purified by a method selected from the group consisting of:

(1) contacting the cyclosiloxane precursor with an adsorbent bed material, so as to remove therefrom at least a portion of the water, and optionally at least impurity, selected from the group consisting of acidic and basic impurities, to produce a cyclosiloxane precursor having a reduced level of water and optionally acidic impurity; and removing the purified cyclosiloxane precursor from the adsorbent bed material; and

(2) distilling a starting mixture comprising at least water and at least one $[\text{SiO}]_n$ cyclosiloxane CVD precursor, in the presence of an azeotropic component, so as to form an azeotropic mixture with the water contained in said starting mixture; in order to produce (A) a distillate fraction comprising water and the azeotropic component and (B) a balance fraction comprising a purified cyclosiloxane precursor, whereby said balance fraction (B) is substantially reduced in water relative to said starting mixture; and

(3) a combination of 1 and 2.